

इंटरनेट

मानक

Disclosure to Promote the Right To Information

Whereas the Parliament of India has set out to provide a practical regime of right to information for citizens to secure access to information under the control of public authorities, in order to promote transparency and accountability in the working of every public authority, and whereas the attached publication of the Bureau of Indian Standards is of particular interest to the public, particularly disadvantaged communities and those engaged in the pursuit of education and knowledge, the attached public safety standard is made available to promote the timely dissemination of this information in an accurate manner to the public.

“जानने का अधिकार, जीने का अधिकार”

Mazdoor Kisan Shakti Sangathan

“The Right to Information, The Right to Live”

“पुराने को छोड़ नये के तरफ”

Jawaharlal Nehru

“Step Out From the Old to the New”

IS 4272 (1986): Vanillin [PCD 18: Natural and Synthetic
Fragrance Materials]



“ज्ञान से एक नये भारत का निर्माण”

Satyanarayan Gangaram Pitroda

“Invent a New India Using Knowledge”



“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

BLANK PAGE



IS : 4272 - 1986

Indian Standard

SPECIFICATION FOR VANILLIN

(First Revision)

UDC 664.571

© Copyright 1987

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR VANILLIN

*(First Revision)*Natural and Synthetic Perfumery Materials
Sectional Committee, PCDC 18*Chairman*

SHRI SHIAM BEHARI LAL

*Members*SHRI SUDHIR JAIN (*Alternate to*
Shri Shiam Behari Lal)

DR AKHTAR HUSAIN

DR C. K. ATAL

DR K. L. DHAR (*Alternate*)
PROF S. C. BHATTACHARYYA
CHIEF CHEMISTDEPUTY CHIEF CHEMIST (*Alternate*)
SHRI A. K. DASSHRI S. K. LUTHRA (*Alternate*)
SHRI B. L. GOPALAKRISHNADR K. B. PATIL (*Alternate*)
DR S. S. GOTHOSKARSHRI A. K. SHRIVASTAVA (*Alternate*)
DR RAJENDRA GUPTADR M. L. MAHESHWARI (*Alternate*)
DR S. K. JAINSHRI V. MUDGAL (*Alternate*)
SHRI J. C. KATHRANISHRI AVINASH K. MARATHE (*Alternate*)*Representing*

Gupta and Co (P) Ltd, Delhi

Central Institute of Medicinal and Aromatic
Plants (CSIR), Lucknow
Regional Research Laboratory (CSIR,)
Jammu (Tawi)Bose Institute, Calcutta
Central Revenues Control Laboratory, New
DelhiDirectorate General of Technical Development,
New DelhiKarnataka Soaps and Detergents Ltd (Soap
and Detergent Division), BangaloreIndian Pharmacopoeia Committee (Ministry
of Health and Family Welfare), New DelhiNational Bureau of Plant Genetic Resources,
New Delhi

Botanical Survey of India, Calcutta

Perfumes and Flavours Association of India,
Bombay*(Continued on page 2)*

© Copyright 1987

BUREAU OF INDIAN STANDARDS

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(Continued from page 1)

<i>Members</i>	<i>Representing</i>
DR S. U. KULKARNI	Hindustan Lever Ltd, Bombay
DR P. CHAKRABORTY (<i>Alternate</i>)	
SHRI G. K. LUTHRIA	Mysore Sugandhi Dhoop Factory Ltd, Bombay
SHRI P. R. MALHANI	The Development Commissioner (Small Scale Industries), New Delhi
DR S. K. KAPOOR (<i>Alternate</i>)	
PROF H. H. MATHUR	Indian Institute of Technology, Bombay
PROF G. K. TRIVEDI (<i>Alternate</i>)	
DR R. K. MATHUR	Regional Research Laboratory (CSIR), Jorhat
DR B. K. SAIKIA (<i>Alternate</i>)	
SHRI C. K. MEHROTRA	Export Inspection Council of India, Calcutta
SHRI S. S. CHOPRA (<i>Alternate</i>)	
DR S. K. MUKHERJEE	Indian Agricultural Research Institute, New Delhi
DR N. K. ROY (<i>Alternate</i>)	
SHRI T. S. NARAYANASWAMY	Swastik Households & Industrial Products Ltd, Bombay
DR SHRIDHAR GOPAL PATNEKAR	Industrial Perfumes Ltd, Bombay
DR ASHOK DIGAMBER WAGH (<i>Alternate</i>)	
SHRI B. K. PATNEY	Richardson Hindustan Ltd, Bombay
SHRI G. S. RANADE (<i>Alternate</i>)	
DR J. S. PRUTHI	Byford Ltd, New Delhi
SHRI P. B. RAO	The Tata Oil Mills Co Ltd, Bombay
SHRI V. VAIDYANATHAN (<i>Alternate</i>)	
SHRI S. S. REVADI	Karnataka Soaps and Detergents Ltd (Sandalwood Oil Division), Mysore
SHRI B. L. GOPALAKRISHNA (<i>Alternate</i>)	
SHRI S. C. SETHI	National Chemical Laboratory (CSIR), Pune
DR A. S. RAO (<i>Alternate</i>)	
SHRI N. B. SHANKARACHARYA	Central Food Technological Research Institute (CSIR), Mysore
DR K. N. GURUDUTT (<i>Alternate</i>)	
SHRI S. A. MOHAMMAD SHAREEF	Maschmeijer Aromatics (India) Ltd, Madras
SHRI M. P. SHIVA	Forest Research Institute & Colleges, Dehra Dun
SHRI G. S. SHUKLA	Directorate of Marketing and Inspection, Faridabad
SHRI R. J. VERMA (<i>Alternate</i>)	
DR A. P. SINGH	Camphor and Allied Products Ltd, Bareilly
SHRI S. N. MEHRA (<i>Alternate</i>)	
SHRI I. SUNDARESH	Basic Chemicals, Pharmaceuticals and Cosmetic Export Promotion Council, Bombay
SHRI Y. G. VAIDYA	Naarden (India) Ltd, Bombay
SHRI S. B. SULE (<i>Alternate</i>)	
SHRI R. V. VAZE	S. H. Kelkars Co Ltd, Bombay
SHRI M. S. SAXENA, Director (P & C)	Director General, BIS (<i>Ex-officio Member</i>)

Secretary

DR (SHRIMATI) VIJAY MALIK
Assistant Director (P & C), BIS

(Continued on page 12)

Indian Standard

SPECIFICATION FOR VANILLIN

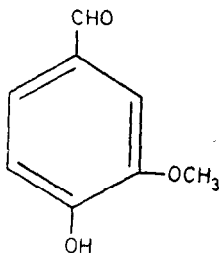
(*First Revision*)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 15 September 1986, after the draft finalized by the Natural and Synthetic Perfumery Materials Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 This standard was first published in 1967. The Sectional Committee responsible for its preparation felt that it should be revised with a view to bring it in line with the trade practices in perfumery technology and also to align with the quality level of the material currently produced and sold in the country.

0.3 Vanillin ($C_8H_8O_3$) is the chief odoriferous principle of vanilla beans from *Vanilla* spp., fam. Orchidaceae. It also occurs in gum benzoin, Peru balsam, etc, in small quantities or is prepared synthetically, mainly from lignin. Vanillin is not only used for flavouring purposes but is also widely employed in many perfumes for its strong and pleasant aroma. Vanillin has also become important as a deodorant to mask the unpleasant odour in many manufactured goods for deodorizing drinking water with an objectionable chlorine odour, for deodorizing rubber goods and for imparting a pleasant odour to wearing apparel. It has the following structural formula.



Vanillin (Molecular Mass 152.14)

0.4 A new requirement of vanillin, percent by mass, minimum along with the gas chromatographic method for determination of vanillin has been included in this revision based on data generated through indigenous testing.

0.5 In the preparation of this standard, considerable assistance has been derived from the Givaudan Index 1961. Ed 2. Givaudan-Delawanna Inc, New York.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for vanillin.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions in IS : 6597-1972† shall apply.

3. REQUIREMENTS

3.1 Description

3.1.1 The material shall consist of white to creamy-white crystalline needles or powder, free from foreign matter and adulterants.

3.1.2 *Reaction with Lead Acetate* — To 1 ml of a saturated aqueous solution of the material, add 1 ml of lead acetate solution 10 percent (w/v); a white precipitate is produced which is soluble in hot water, but separates in scales on cooling.

*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to natural and synthetic perfumery materials

3.1.3 Reaction with Ferric Chloride — To 10 ml of a saturated solution of the material, add 3 to 5 drops of ferric chloride solution. A blue colour is produced which becomes brown on heating the solution 80°C for a few minutes. On cooling, a white or almost white precipitate is produced.

3.2 Solubility — The material shall be clearly soluble in three volumes of ethyl alcohol (70 percent by volume) when tested as prescribed in IS : 326 (Part 6)-1986*.

3.3 The material shall also be tested olfactorily and specially for by-notes as prescribed under 4 and 5 of IS : 2284-1963†.

3.4 The material shall also comply with the requirements given in Table 1.

4. PACKING AND MARKING

4.1 The material shall be packed in amber-coloured glass bottles or in fibre board boxes with suitable polyethylene liner (*see* IS : 2508-1984‡), as agreed to between the purchaser and the supplier.

TABLE 1 REQUIREMENTS FOR VANILLIN
(*Clauses 3.4 and 6.1*)

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN	
			Appendix	IS : 2284-1963*
(1)	(2)	(3)	(4)	(5)
i)	Odour	Strong, persistent, suggestive of vanilla beans	—	4 and 5
ii)	Melting range, °C	81-83	A	—
iii)	Sulphated ash, percent by mass, <i>Max</i>	0.1	B	—
iv)	Vanillin percent by mass, <i>Min</i>	98	C	—

*Method for olfactory assessment of natural and synthetic perfumery materials.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 6 Determination of solubility in ethanol (*second revision*).

†Method for olfactory assessment of natural and synthetic perfumery materials.

‡Specification for low density polyethylene films (*second revision*).

4.2 The particular source from which the material has been obtained shall be marked on each container.

4.3 The material shall be protected from light and stored in a cool and dry place.

4.4 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

5. SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in IS : 326 (Part 1)-1984*.

5.2 Number of Tests — Tests for determination of all the characteristics shall be conducted on the composite sample.

5.3 Criteria for Conformity — The lot shall be considered as conforming to the specification if the composite sample satisfies all the requirements specified in this standard.

6. TEST METHODS

6.1 Tests shall be conducted as prescribed under 3.1, 3.2, 3.3 and the appropriate references specified in col 4 and 5 of Table 1.

6.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (see IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and test for natural and synthetic perfumery materials: Part 1 Sampling (*second revision*).

†Specification for water for general laboratory use (*second revision*).

APPENDIX A

[Table 1, item (ii)]

DETERMINATION OF MELTING POINT

A-0. GENERAL

A-0.1 Outline of the Method — The temperature at which the material melts and becomes liquid throughout as shown by the formation of a definite meniscus is determined as the melting point of the material.

A-1. APPARATUS

A-1.1 Capillary Tube — of glass, closed at one end; thickness of the wall about 0.10 to 0.15 mm; length suitable for the apparatus used and of 0.9 to 1.1 mm internal diameter.

A-1.2 Thermometers — accurately calibrated, covering the range from -10°C to $+110^{\circ}\text{C}$, the length of two deg on the scale being not less than 0.8 mm. These thermometers shall be of mercury-in-glass, solid stem type; the bulb shall be cylindrical in shape and made of thermometric glass suitable for the range of temperature covered. Each thermometer shall be fitted in a safety case.

A-1.3 Glass Heating Vessel — of suitable construction and capacity fitted with a stirring device capable of rapidly mixing the liquid.

A-2. PROCEDURE

A-2.1 In an evaporating dish, spread a small quantity of the finely powdered material in a thin layer and dry at a temperature below its melting point or in a vacuum desiccator over sulphuric acid for 24 hours.

A-2.2 Transfer a quantity of the dried powder to a dry capillary tube and pack the powder by tapping the tube on a hard surface so as to form a tightly packed column of 2 to 4 mm in height. Attach the capillary tube and its contents to the thermometer so that the closed end is at the level of the middle of the bulb and heat in the heating vessel fitted with an auxiliary thermometer, regulating the rise of temperature during the first period to $3^{\circ}\text{C}/\text{min}$. When the temperature reached is 10°C below the lowest figure of the range for the material being tested, the heating of the vessel is adjusted so that the rate of rise in temperature is 1 to $2^{\circ}\text{C}/\text{min}$.

A-2.3 Note the temperature reading at which liquefaction of the material occurs as indicated by the formation of a definite meniscus. Record the temperature reading as the melting point of the material.

APPENDIX B

[Table 1, item (iii)]

DETERMINATION OF SULPHATED ASH

B-0. GENERAL

B-0.1 Outline of the Method — The material is incinerated in presence of concentrated sulphuric acid and the resulting ash estimated.

B-1. REAGENTS

B-1.1 Concentrated Sulphuric Acid — conforming to IS : 266-1977*.

B-2. PROCEDURE

B-2.1 Weigh accurately about 10 g of the material in a tared silica dish of about 20 ml capacity. Add about 0.5 ml of concentrated sulphuric acid and ignite gently. Cool in a desiccator and weigh. Repeat this process till successive weighings agree within 1 mg.

B-3. CALCULATION

B-3.1 Calculate sulphated ash as follows:

$$\text{Sulphated ash, percent by mass} = 100 \frac{m}{M}$$

where

m = mass in g of the residue, and

M = mass in g of the material taken for the test.

*Specification for sulphuric acid (*second revision*).

APPENDIX C

[Table 1, item (iv)]

GAS CHROMATOGRAPHIC ANALYSIS FOR DETERMINATION OF VANILLIN

C-0. GENERAL

C-0.1 The chromatographic conditions given here are for guidance only.

C-0.2 Outline of the Method — A sample of the material is dissolved in a suitable solvent (for example, cyclohexane and diethyl ether) and is injected into the gas chromatograph where it is carried by the carrier gas from one end of the column to the other. During its movement, constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of the column one after another and are detected by suitable means whose response is related to the amount of a specific component leaving the column.

C-1. APPARATUS

C-1.1 Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. The typical chromatograph for vanillin using a chromatograph with the following chromatographic conditions is shown in Fig.1.

- | | |
|----------------------------------|-----------------------------------|
| a) <i>Sample:</i> | Vanillin |
| 1) Material | Copper |
| 2) Length | 6 m |
| 3) Outer diameter (OD) | 0.635 cm |
| 4) Inner diameter (ID) | 0.476 cm |
| 5) Stationary phase | Carbowax 20 M, 10 percent by mass |
| 6) Solid support | Chromosorb WAW 60-80 mesh |
| b) <i>Carrier Gas</i> | Nitrogen |
| c) <i>Conditions :</i> | |
| 1) Column temperature isothermal | 205°C |
| 2) Injection port temperature | 200°C |
| 3) Carrier gas flow | 20 ml/min |
| 4) Inlet pressure | 3.5 kg/cm ² |

d) *Detector:*

- | | |
|----------------|-------|
| 1) Type | F.I.D |
| 2) Temperature | 280°C |

e) *Recorder:*

- | | |
|----------------|-------------|
| 1) Span | 1 mV |
| 2) Chart speed | 0.25 cm/min |

- | | |
|-----------------------|----|
| f) <i>Attenuation</i> | 64 |
|-----------------------|----|

NOTE — This analysis may also be accomplished with columns containing carbowax — 20 M, D. G. S (Diethylene Glycol Succinate).

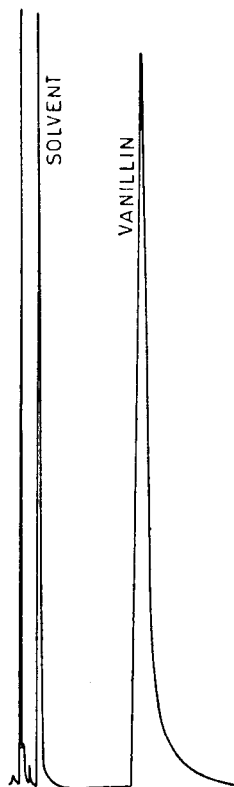


FIG. 1 TYPICAL CHROMATOGRAM OF VANILLIN

C-2. PROCEDURE

C-2.1 Conduct the flow of the carrier gas and inject the sample (dissolved in the suitable solvent) at inject port where it is vaporized and well mixed with the carrier. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. As the different constituents pass through the detector, they give signals corresponding to the amount of particular constituents leaving the column. The detector signals, on transmission to the recorder, plots the chart. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

NOTE — For separation to be efficient, it is necessary that the column is maintained at the temperature suggested throughout the time requirement for the resolution of the constituents.

C-3. CALCULATION

C-3.1 Area Measurement (see Note 1) — Since normal peaks approximate a triangle, the area is measured by multiplying the peak height with the width of half-height. The normal peak base is not taken since large deviations may be observed due to tailing or adsorption. This technique is rapid, simple and fairly accurate when peaks are symmetrical and of reasonable width.

C-3.2 Area Normalization (see Note 2) — By normalizing, it is meant, calculating the percentage composition by measuring the area of each and dividing the individual areas by total area, for example,

$$\text{Percentage of } A = \frac{\text{Area of } A}{\text{Total area}} \times 100$$

NOTE 1 — Other methods of area measurement namely, triangulation, disc integrator and electronic digital integrator, if fixed with GLC machine, would be of great advantage.

NOTE 2 — Internal standardization can be used if pure appropriate internal standard is available. This method is known as relative or indirect calibration.

(Continued from page 2)

Synthetic Perfumery Materials Subcommittee, PCDC 18 : 2

Convener

SHRI SUDHIR JAIN

Representing

Gupta and Co (P) Ltd, Delhi

Members

SHRI SHIAM BEHARI LAL (*Alternate to*

Shri Sudhir Jain)

SHRI R. C. BASSI

The Indian Turpentine and Rosin Co Ltd,
Bareilly

SHRI S. C. SAKSENA (*Alternate*)

SHRI A. K. DAS

Directorate General of Technical Development,
New Delhi

SHRI S. K. LUTHRA (*Alternate*)

SHRI B. HANUMANTHA RAO

Bush Boake Allen (India) Ltd, Madras

SHRI B. K. BALIGA (*Alternate*)

SHRI B. T. JANARDHAN

Karnataka Soaps and Detergents Ltd (Sandal-
wood Oil Division), Mysore

DR K. B. PATIL (*Alternate*)

SHRI G. D KELKAR

S. H. Kelkar and Co Pvt Ltd, Bombay

DR S. U. KULKARNI

Hindustan Lever Ltd, Bombay

SHRI S. KRISHNAMOORTHY (*Alternate*)

DR U.R. NAYAK

National Chemical Laboratory (CSIR), Pune

DR A. S. RAO (*Alternate*)

DR SHRIDHAR GOPAL PATNEKAR

Industrial Perfumes Ltd, Bombay

DR ASHOK DIGAMBER WAGH (*Alternate*)

SHRI P. B. RAO

Tata Oil Mills Co Ltd, Bombay

DR A. P. SINGH

Camphor and Allied Products Ltd, Bareilly

SHRI S. N. MEHRA (*Alternate*)

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 331 01 31, 331 13 75

Telegrams: Manaksanstha
(Common to all Offices)

Regional Offices:

Telephone

*Western : Manakalaya, E9 MIDC, Marol, Andheri (East), BOMBAY 400093 6 32 92 95

†Eastern : 1/14 C.I.T. Scheme VII M, V.I.P. Road, Maniktola, CALCUTTA 700054 36 24 99

Northern : SCO 445 - 446, Sector 35 - C, CHANDIGARH 160036 { 2 18 43
3 16 41

Southern : C.I.T. Campus, MADRAS 600113 { 41 24 42
41 25 19
41 29 16

Branch Offices:

'Pushpak', Nurmohamed Shaikh Marg, Khanpur, AHMADABAD 380001 { 2 63 48
2 63 49

'F' Block, Unity Bldg, Narasimharaja Square, BANGALORE 560002 22 48 05

Gangotri Complex, Bhadbhada Road, T. T. Nagar, BHOPAL 462003 6 67 16

Plot No. 82/83, Lewis Road, BHUBANESHWAR 751002 5 36 27
53/5 Ward No. 29, R.G. Barua Road, 5th By-lane, GUWAHATI 781003 —

5-8-56C L. N. Gupta Marg, HYDERABAD 500001 23 10 83

R14 Yudhister Marg, C Scheme, JAIPUR 302005 { 6 34 71
6 98 32

117/418 B Sarvodaya Nagar, KANPUR 208005 { 21 68 76
21 82 92

Patliputra Industrial Estate, PATNA 800013 6 23 05

Hantex Bldg (2nd Floor), Railway Station Road, TRIVANDRUM 695001 7 66 37

Inspection Office (With Sale Point):

Institution of Engineers (India) Building, 1332 Shivaji Nagar, PUNE 411005 5 24 35

*Sales Office in Bombay is at Novelty Chambers, Grant Road, Bombay 400007 89 65 28

†Sales Office in Calcutta is at 5 Chowringhee Approach, P.O. Princep Street, Calcutta 700072 27 68 00